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**Abstract.** Macrocyclic compounds such as crown ethers and their analogs are able to selectively bind metal ions due to donor atoms in the ring, which makes them promising for the development of sorbents. Polymers with nitrogen- and sulfur-containing crown ethers are of interest because of their high selectivity and stability. The aim of this work is to synthesize sorbents based on aza- and thiacrown-ether compounds forming selective complexes with metal ions in solution. The synthesis, structural characterization and sorption properties of polymeric sorbents based on macrocyclic compounds containing nitrogen and sulfur atoms - aza- and thiacraun- esters - are considered in this work. The work focuses on the advantages of immobilization of macrocyclic ligands on a polymer matrix in order to increase their stability, reduce toxicity and improve sorption characteristics. Immobilization was carried out on a chloromethylated copolymer of styrene and divinylbenzene by nucleophilic substitution in dimethylformamide. Various crown ether derivatives were successfully anchored: monoaza-15-crown-5, diaza-18-crown-6. The structural changes were confirmed by IR spectroscopy and optimal reaction conditions in terms of temperature, time and reagent ratio were established. The obtained polymers were investigated for their ability to sorb metal ions in aqueous and methanol media. The sorption efficiency was found to depend significantly on the structure of the macrocycle, especially on the number of sulfur atoms, as well as on the nature of the solvent. Polymers containing DADT18K6 and DATT18K6 showed the highest affinity to $Hg^{2+}$ and $Pb^{2+}$ ions. The obtained results confirm the promising application of immobilized aza- and thiacraun ether sorbents for selective extraction of metal ions.

**Keywords:** crown ethers, aza- and thiacraun ether compounds, polymeric sorbents, complexation with metal ions, selective sorption, immobilization

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# КРАУН-ЭФИРЛЕР НЕГІЗІНДЕГІ ЖАҢА ТАЛҒАМДЫ СОРБЕНТТЕРДІҢ СИНТЕЗІ МЕН СОРБЦИЯЛЫҚ ҚАСИЕТТЕРІ

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**Аннотация.** Краун-эфирлер мен олардың аналогтары сақина құрылымындағы донорлық атомдар арқылы металл иондарын таңдамалы байланыстыру қабілетіне ие, бұл оларды тиімді сорбенттер жасау үшін перспективалы етеді. Әсіресе азот пен күкірт атомдарын қамтитын краун-эфирлер негізіндегі полимерлер жоғары талғамдылық және тұрақтылық көрсетеді. Осы жұмыстың мақсаты – аза - және тиакраун – эфирлі қосылыстардың негізінде, ерітіндідегі металлл иондарымен селективті комплекс түзетін сорбенттерді синтездеу болып табылады. Бұл жұмыста құрамында азот және күкірт атомдары бар макроциклді қосылыстар – аза- және тиакроунды эфирлер негізіндегі полимерлік сорбенттердің синтезі, құрылымдық сипаттамалары және сорбциялық қасиеттері қарастырылады. Бұл қосылыстар сақина құрылымында донор атомдарының (азот, оттегі және күкірт) болуына байланысты металл иондарымен тұрақты және таңдамалы кешендер түзу қабілетіне ие. Жұмыста олардың тұрақтылығын арттыру, уыттылығын төмендету және сорбциялық сипаттамаларын жақсарту мақсатында макроциклді лигандтарды полимерлі матрицада иммобилизациялаудың артықшылықтарына назар аударылады. Иммобилизация диметилформамидті ортада нуклеофильді алмастыру арқылы стирол мен дивинилбензолдың хлорметилденген сополимерінде жүргізілді. Әртүрлі кроундық эфир туындылары: моноаза-15-краун-5 (MA15K5), диаза-18-краун-6 (DA18K6), сондай-ақ аралас аза-тиакроунды эфирлер (DADT18K6 және DATT18K6) сәтті бекітілді. Құрылымдық өзгерістер ИҚ-спектроскопиялық әдістермен расталды, температура, уақыт және реагент қатынасы бойынша оңтайлы реакция жағдайлары белгіленді. Алынған полимерлер су және метанол орталарында металл иондарын ($Na^+$, $K^+$, $Ag^+$, $Hg^{2+}$, $Pb^{2+}$) сорбциялау қабілеті зерттелді. Сорбцияның тиімділігі макроцикл құрылымына, атап айтқанда күкірт атомдарының санына, сондай-ақ еріткіштің табиғатына айтарлықтай тәуелді екендігі анықталды. Құрамында DADT18K6 және DATT18K6 бар полимерлер $Hg^{2+}$ және $Pb^{2+}$ иондары үшін ең жоғары жақындықты көрсетті. Алынған нәтижелер металл иондарын таңдамалы экстракциялау және қоршаған ортаны қорғау міндеттері үшін иммобилизацияланған аза- және тиакроун-эфир сорбенттерін пайдалану мүмкіндігін растайды.

**Түйін сөздер:** краун-эфирлер, аза - және тиакраун – эфирлі қосылыстар, полимерлі сорбенттер, металл иондарымен кешентүзу, таңдамалы сорбция, иммобилизация

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# СИНТЕЗ И СОРБЦИОННЫЕ СВОЙСТВА НОВЫХ СЕЛЕКТИВНЫХ СОРБЕНТОВ НА ОСНОВЕ КРАУН-ЭФИРОВ

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**Аннотация**. Макроциклические соединения, такие как краун-эфиры и их аналоги, способны избирательно связывать ионы металлов за счёт донорных атомов в кольце, что делает их перспективными для создания сорбентов. Особенно интересны полимеры с азот- и серусодержащими краун-эфирами, так как они обладают высокой селективностью и стабильностью. Целью данной работы является синтез сорбентов на основе аза- и тиакраун-эфирных соединений, образующих селективные комплексы с ионами металлов в растворе. В работе рассмотрены синтез, структурная характеристика и сорбционные свойства полимерных сорбентов на основе макроциклических соединений, содержащих атомы азота и серы - аза- и тиакраун-эфиров. Эти соединения обладают способностью образовывать устойчивые и селективные комплексы с ионами металлов благодаря наличию донорных атомов (азота, кислорода и серы) в их кольцевой структуре. Работа акцентирует внимание на преимуществах иммобилизации макроциклических лигандов на полимерной матрице с целью повышения их стабильности, снижения токсичности и улучшения сорбционных характеристик. Иммобилизацию проводили на хлорметилированном сополимере стирола и дивинилбензола посредством нуклеофильного замещения в среде диметилформамида. Были успешно закреплены различные производные краун-эфиров: моноаза-15-краун-5, диаза-18-краун-6, смешанные аза-тиакраун-эфиры. Структурные изменения подтверждены методами ИК-спектроскопии, установлены оптимальные условия реакции по температуре, времени и соотношению реагентов. Полученные полимеры исследовали на способность сорбировать ионы металлов ($Na^+$, $K^+$, $Ag^+$, $Hg^{2+}$, $Pb^{2+}$) в водной и метанольной среде. Установлено, что эффективность сорбции существенно зависит от структуры макроцикла, в особенности от числа атомов серы, а также от природы растворителя. Полимеры, содержащие ДАДТ18К6 и ДАТТ18К6, показали наибольшую аффинность к ионам $Hg^{2+}$ и $Pb^{2+}$. Полученные результаты подтверждают перспективность применения иммобилизованных аза- и тиакраун-эфирных сорбентов для селективного извлечения ионов металлов и в задачах охраны окружающей среды.

**Ключевые слова:** краун-эфиры, аза- и тиакраун-эфирные соединения, полимерные сорбенты, комплексообразование с ионами металлов, селективная сорбция, иммобилизация

**Introduction.** The use of synthetic macrocyclic compounds (crown ethers and cryptands) and their acyclic analogs (podants) as complexing agents of cationic, anionic and neutral low molecular weight substances is of great scientific and practical interest. At present, many new macrocyclic compounds have been synthesized, and their application is significantly expanding (Dovhyi, 2017; Hiraoka, 2016; Basok, 2021; Gokel, 2016; Guo, 2014).

A characteristic feature of this class of compounds is the ability to form complexes due to the unshared pairs of electrons of the valence shell of oxygen, nitrogen and sulfur atoms in the ring of the crown ether molecule, directed to the center of the ring. Crown ether compounds have the “ marvelous” property of forming strong, elegant complexes with metal cations in solution.

The ability of crown ether compounds to selectively interact with metal ions underlies their practical applications. The selectivity of compounds depends on the structure of ligands in the molecule, the presence of functional groups and donor atoms in the molecule. It is known that high selectivity of reagents depends not on the nature of donor atoms in the cycle, but on the stability of the structure of this cycle (Yakshin, 2010; Fegtle, 1988; Tsivadze, 1991; Li, 2017).

In the last 15-20 years, the presence of heteroatoms in the macrocycle, such as oxygen atoms, as well as other sulfur and nitrogen atoms (thia- and azacrown compounds) has also attracted much interest (Schneider, 2020; Zakurdaeva, 2010; Gokel, 2013).

The presence of such heteroatoms in the molecular ring ensures the compatibility of donor atoms and metal atoms in the macrocycle, as well as weak acids and bases (Pearson's concept) (Ergozhin, 1995).

Insoluble polycrown ethers have many advantages over their low molecular weight monomeric counterparts. Insoluble crown ether polymers used as membranes or sorbents are known to be highly effective in the selective separation of metal ions from solution and can be reused many times through recycling. Such polymeric crown ether sorbents are much less toxic and harmful than their corresponding low molecular weight counterparts [Ergozhin, 1994]. Therefore, the demand for polymeric crown ether compounds has increased dramatically in recent years. It was found that when one or more oxygen atoms in the molecular ring are replaced by nitrogen and sulfur atoms, the ability to complex with transition metal ions in solution increases.

However, crown ether polymers containing nitrogen and sulfur atoms in the ring are still not fully understood. Due to these circumstances, the search for new methods for the synthesis of polymeric aza- and thiacraun-ether compounds, the study of their physicochemical and complexing properties is currently one of the most important unsolved problems.

The aim of this work is to synthesize sorbents based on aza- and thiacraun-ether compounds forming selective complexes with metal ions in solution.

## Materials and methods

Crown ethers containing nitrogen and sulfur atoms: monoaza-15-crown-5 (MA15K5), 1,10-diaza-18-crown-6 (DA18K6), 1,10-diaza-4,7-dithia-18-crown-6 (DADT18K6) and 1,10-diaza-4,7,13,16-tetrathia-18-crown-6 (DATT18K6) were obtained by Kellogg's method (Bezhin 2017).

Copolymer of chloromethylated styrene and divinylbenzene - production of Cherkasskiy chemical plant (Ukraine).

Synthesis of polymacrocyclic esters. To obtain high-yield polymers with high sorption capacity and a certain degree of swelling, yellow chloromethylated copolymer of styrene and divinylbenzene (20 g) was placed in a three-well glass flask equipped with a reflux condenser, thermometer and mechanical stirrer, dimethylformamide (10-15 ml) was added and stirred until the polymer swelled. One hour later, after swelling of the polymer, the compound 1,10-diaza-4,7,13,16-tetrathia-18-crown-6 was added to the flask. This mixture in the flask was stirred at 90°C for 6-8 hours at very high speed.

The reaction product was cooled to room temperature, filtered, washed with distilled water, and the resulting polycrown ether compound was dried to constant weight.

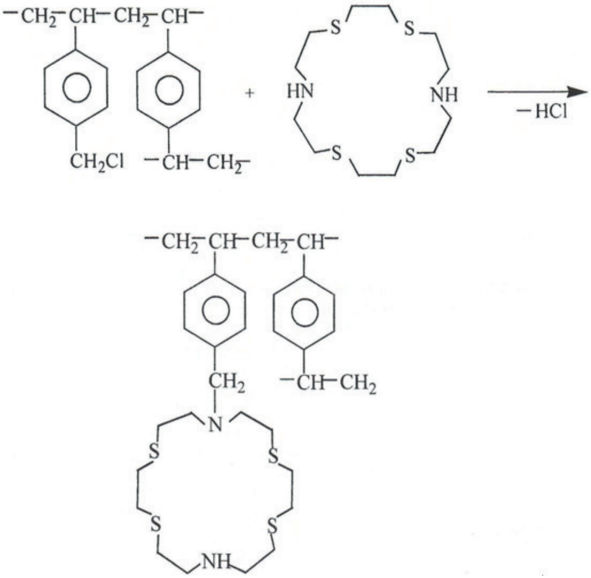
The sorption kinetics was studied using the limited solution volume method (Ergozhin, 2020).

To obtain sorption kinetic curves, 0.1 g of samples (m) of polysaccharide material were placed in test tubes and 10 ml (V) of metal sulfate or chloride at initial concentration ($C_o$) was added, and after 1 hour the mixture was kept at a certain temperature for up to 2 hours, the solution was separated from the sorbent by filtration and the current concentration of metal ions (Cτ) was determined by atomic absorption spectroscopy on a «Saturn» instrument. Sorption capacity (Aτ) of sorbents (at any given time) is calculated by the formula:

$$A_\tau = \frac{(C_0 - C_\tau)}{m} \cdot V.$$

IR spectra were recorded on KBr tablets on a Vector-22 Fourier transform spectrometer.

**Results and discussion.** Immobilization of macrocyclic low molecular weight compounds on polymer carriers is a widely used method in the field of polymer crown ether synthesis. The interaction reaction of a gel-like and porous copolymer of chloromethylated styrene and divinylbenzene (CMS) with the macrocyclic compound 1,10-diaza-4,7,13,16-tetrathia-18-crown-6 (DATT18K6) has been studied for the first time to obtain polymers for this purpose. The reaction proceeds as follows:



Comparison of the IR spectra of aza- and azathiacraun esters, CMS and immobilization products showed that the absorption bands (1270, 970 cm$^{-1}$) of $CH_2Cl$-group are present in the spectrum of chloromethylated polystyrene spatial structure. Absorption bands characteristic of the aromatic ring in the region of 1600 cm$^{-1}$, frequencies of deformation non-planar vibrations of the CH-group of the aromatic ring -700 and 820 cm$^{-1}$ characteristic of the disubstituted benzene ring are also prescribed. In the IR spectrum of azacrown esters immobilized on chloromethylated copolymer of styrene and divinylbenzene, a broad band corresponding to the valence vibrations of the NH group in the region of 3335 cm$^{-1}$ is present. There is a strain vibration frequency of the NH bond at 1590 cm$^{-1}$. A new intense band characteristic of the simple ether bond appears in the spectrum in the region of 1120 cm$^{-1}$, and the intensity of the $CH_2Cl$-group frequency decreases strongly.

The properties of polymers immobilized by crown ether functional groups depend mainly on the degree of incorporation of these functional groups into the polymers, i.e., their amount. The degree of immobilization of the HMS copolymer with crown ether functional groups depends on several factors. The main factors are: purity and monomer ratio, solution concentration, reaction temperature and time, and the nature of the solvent.

The study of the influence of the nature of solvent showed that the highest yields were achieved using DMFA. This can be explained by the fact that in DMFA medium they bind more completely to the polymer matrix than in chloroform. Thus, the yields of crown-ether copolymers are 85% and 56% with the respective participation of 1,10-diaza-18-crown-6 1,10-diaza-4,7,13-tritya15-crown-5 in 5 h at 90$^{o}$C .

In the case of 1,10-diaza-4,7-dithia18-crown-6, the maximum yield of the final product was achieved in 4 h at 90$^{o}$ C . A study of the effect of temperature on the reaction of various azathiacraun esters with CMS showed That the high degree of transformation is achieved at a temperature of 90-95$^{o}$C. Increasing the temperature of the medium above the optimum temperature reduces the mass fraction of nitrogen in the polymer composition, as destructive processes may occur.

The study of the effect of molar ratios of macrocycles with CMS on the yield of the final product shows that the optimal ratios depend on the nature of azathiacraun esters. Immobilized polymers with the structure of 18-crown-6 are formed in high yields. This can be explained by the symmetry of the arrangement of atoms in the ring and the flexibility of this structure. Further increasing the concentration of azathiacraun esters does not increase the mass fraction of nitrogen in the copolymer composition. Thus, the study of the condensation reaction of azathiacrown ethers with CMS in DMFA medium shows that the highest nitrogen content in the polymer composition is achieved at a ratio of reagents 1:1 and up to 1.4 at a temperature of 90-95$^{o}$ C, the process duration of 4-5 h. The yield of the product under these conditions is 85%.

In this work, the features of obtaining immobilized polymers based on aza- and thiacraun esters have been studied. To determine the reactivity of aza- and thiacraun esters, the process was carried out in the polar aproton solvent dimethylformamide. The reaction rate constant and activation energy (15.2-21.0 kJ/mol) were determined. The amount of crown ether groups in the sorbent is 1.06-1.24 mmol/g.

One of the main properties of polymeric crown ether compounds is their stable complexation ability. However, the complexation properties of polymers synthesized on the basis of macrocycles containing nitrogen and sulfur atoms in the molecular ring have not been studied. It was noted above that polymeric sorbents have several advantages over their low molecular weight counterparts.

In this context, we studied the complexation properties of immobilized polyaza- and thiacraun esters with metal ions using the atomic adsorption method. The complex between immobilized macrocyclic ligands and metal cations is formed between negatively charged nitrogen and sulfur electron donor atoms located in the crown ether ring and positively charged metal cations. The degree of selective correspondence of the macrocycle structure in the polymer to the metal ion was determined by the sorption properties of the sorbents in aqueous and methyl alcohol solvents. The sorption capacities of immobilized polyazathiacraun esters are given in Table 1.

Table 1. Sorption data of immobilized polyazathiacraun esters in water and methyl alcohol.

| Based on polycrown ether | Sorption capacity, mg/g (in water/methyl alcohol) | | | | |
|---|---|---|---|---|---|
| | Cations | | | | |
| | $Na^+$ | $K^+$ | $Ag^+$ | $Hg^{2+}$ | $Pb^{2+}$ |
| MA15K5 | 33,15/52,14 | 36,96/54,20 | 48,44/66,96 | 106,14/124 | 110,22/128,34 |
| DA18K6 | 38,44/46,26 | 51,26/68,18 | 46,96/64,26 | 101,3/119 | 106,29/123,16 |
| DADT18K6 | 32,15/48,25 | 60,19/79,28 | 222,25/241,92 | 429,8/448 | 445,53/463,68 |
| DATT18K6 | 32,44/50,92 | 76,44/92,16 | 210,14/228,96 | 404,5/424 | 421,52/438,84 |

Analyzing the quantitative data presented in the table, it follows that the sorption capacity of the sorbents depends on the structure and size of the macrocycles, the nature of the cation and the number of sulfur atoms in the crown ring. Thus, the maximum value of sorption capacity is observed in the case of $Hg^{2+}$ and $Pb^{2+}$ ions with sorbents containing DADT18K6 functional groups. The same sorbents form stable complexes with Ag+ cations. The sorption of the sorbents was found to be higher in organic solvents compared to water. It is also known from these studies that the nature of solvents has a significant effect on the solvation of complexes.

**Conclusion.** As a result of the studies, polymeric sorbents based on aza- and thiacraun ethers with high selectivity to metal ions were synthesized. The method of immobilization of low-molecular macrocyclic compounds on the polymer matrix allowed to obtain stable sorbents with high sorption capacity and thermal stability. It was found that the structure and size of the macrocycle, as well as the number of sulfur atoms in the ring have a significant influence on the sorption properties. The highest sorption capacity was observed in interaction with heavy metal ions such as $Hg^{2+}$ and $Pb^{2+}$, especially for polymers with DADT18K6 functional groups. It was also observed that sorption in organic solvents was higher than in water, indicating the influence of the medium on the complexation process. The obtained data confirm the promising use of polymeric aza- and thiacraunic esters as selective sorbents for the extraction of metal ions from solutions.

Polycrown ethers containing nitrogen and sulfur atoms in the macrocycle of the molecule can be used as sorbents for selective separation of transition metal ions close to each other and as catalysts of phase transitions.

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